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THE EFFECTS OF PREOXIDATION TREATMENTS ON THE SPECTRAL NORMAL

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TYPE 347 STAINLESS STEEL AT TEMPERATURES OF

900°, 1,200°, 1,500°, and 1,800° F

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THE EFFECTS OF PREOXIDATION TREATMENTS ON THE SPECTRAL NORMAL
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ABSTRACT

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The spectral normal emittance of oxidized Inconel, Inconel-X, and type 347 stainless steel was determined at temperatures of 900°, 1,200°, 1,500°, and 1,800° F over a wavelength range of 1 through 15 microns. Polishing, grit blasting, etching, or combinations of these were used as preoxidation treatments. Large effects of variations in oxidation times and preoxidation treatments were found.

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INTRODUCTION

A considerable quantity of emittance data is available for use by designers, but for many materials there are large differences between the data obtained by different investigators testing nominally the same material. The variations arise from problems inherent in the methods of measurement, unknowns in the composition of materials, contamination of surfaces, and varying techniques for preparation of the surfaces. The most common source of discrepancies appears to be the last of these, namely, the lack of accurately defined, reproducible surfaces whose preparation and method of test is clearly and accurately described. Information on the reproducibility of a surface, specifications describing, in detail, the exact surface preparation procedures, and a clearly defined and reliable test procedure are necessary in order for designers to be able to consider a material and predict its usefulness.

At the Langley Research Center of the NASA we have investigated the relation between emittance and the method of preparing the surface for three oxidized metal surfaces. The three metals were Inconel, Inconel-X, and type 347 stainless steel. Four basic types of surface preparation procedures were used prior to oxidation; the resulting surface conditions are denoted as "as received," "etched," "grit blasted," and "polished." These preoxidation treatments are

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described in detail in tables I and II. Other surfaces were formed by combining or varying these treatments or the oxidation time. Thus, correlation of emittance characteristics with surface preparation procedure was obtained for quite a large range of surface preparation procedures. Data were also obtained on the reproducibility of the oxidized surfaces. Each specimen was tested by measuring the spectral normal emittance from 1.0 to 15.0 microns at four temperatures, 900°, 1,200°, 1,500°, and 1,800° F. Spectral rather than total emittance measurements were made in order to provide more insight into the effects of surface preparation on the emittance of these oxidized alloys.

APPARATUS

The apparatus and procedures used in the performance of this study are completely described in reference 1. A McMahon type blackbody furnace, reference 2, was used for heating the test specimens during the emittance measurements. A specially modified spectrophotometer with a sodium chloride prism and high-speed thermocouple detector were used to obtain these measurements.

SPECIMENS AND PREPARATION PROCEDURES

The specimens used in this study were 4-inch-diameter semicircles 1/16 inch thick.

Several oxidized surfaces were prepared on each of the three materials, Inconel, Inconel-X, and type 347 stainless steel. The preoxidation treatments, which are described in tables I and II, are designated as follows: as received (AR), etched (E_1) or (E_2), grit blasted (GB_1) or (GB_2), and polished (P). Other surfaces were formed by combining these preoxidation treatments or changing the oxidation time. The exact method of preparing these test specimens will be explained in detail in the following sections.

Degreasing

In general, the specimens were subjected to an initial degreasing. The procedure consisted of a thorough swabbing with methyl alcohol, using cotton swabs, then with deionized water, then with reagent grade acetone, and finally with deionized water. The specimens were then air dried. If water spots appeared on the specimens after drying, the process was repeated. Rubber gloves were used while preparing these specimens, and cotton gloves were used in handling the specimens between stages of preparation and testing, in order to avoid contamination from body oils or fingerprints. As will be described later, a different degreasing process was used for some of the specimens.

Oxidation

All specimens were oxidized in a clean electric furnace preheated to the desired oxidation temperature. The specimens were oxidized for the time and at the temperature required to provide the oxide surface desired for testing. In general, the oxidation temperature and time for Inconel and Inconel-X were 2,000° F and 20 minutes, designated as O₁, in table II; for type 347 stainless steel they were 1,800° F and 20 minutes, designated as O₂ in table II. Some Inconel specimens were given longer oxidation times.

As Received

Those surfaces designated AR were given only the degreasing treatment before being oxidized. Otherwise, they were essentially as received from the supplier.

Etched

Surfaces designated E were, after degreasing, placed in an etching solution. The specimens designated E₁ were etched by a solution composed of 32 parts reagent grade nitric acid, 70 percent HNO₃, 8 parts reagent grade hydrofluoric acid, 48 percent HF, and 32 parts deionized water. The etching time was 1½ hours. Upon removal from the solution the specimens were rinsed with deionized water, swabbed with methyl alcohol using cotton swabs, and rinsed with deionized water again and air dried.

This etching process (E₁) was used for the Inconel and Inconel-X specimens. These specimens were oxidized at 2,000° F for a period of 20 minutes.

The specimens designated (E₂) were etched by a solution composed of 100 ml of hydrochloric acid, 36.5 to 38.0 percent HCL, 25 grams of powdered reagent grade chromic acid, 100 percent CrO₃, and 50 ml of deionized water. The etching time varied for this particular etching solution. A period of 5 minutes was allowed for the first two specimens and the time was decreased to 3 minutes as more specimens were etched in the same solution. (It was found that the action of the solution upon the surface of the stainless steel became more rapid with use.) The etched specimen was removed from the etching solution and immediately immersed in deionized water to rinse the residue left by the etching solution from the specimen. The specimens were then swabbed with cotton swabs in hot deionized water, then swabbed with methyl alcohol and rinsed with deionized water and air dried. This etching process was used on type 347 stainless-steel specimens only. These specimens were oxidized at 1,800° F for a period of 20 minutes (O₂).

Polished

Surfaces designated P were degreased by the standard procedure and then hand polished through four papers, 240, 320, 400, and 600 grit. This process gives a flat, smooth surface, although it is not very highly polished. The specimens were again cleaned by the standard procedure to remove all contaminants from the polishing process, and finally oxidized as previously described.

Surfaces designated GB were first degreased in a vapor degreaser using trichloroethylene as the solvent. This was done to remove oils and other surface contaminants before blasting the surface. The specimens designated GB₁ were blasted with number 100 silicon carbide grit under 90 psi at a distance of 6 inches. They were then cleansed again, using the vapor degreaser, to remove all fingerprints and contamination from the blasting process and were then air dried.

The surface GB₁ was prepared on all three alloys, Inconel, Inconel-X, and type 347 stainless steel. The Inconel and Inconel-X specimens were oxidized at 2,000° F for 20 minutes while the stainless steel was oxidized for 20 minutes at 1,800° F.

The specimens designated GB₂ were prepared in the same manner as the GB₁ specimens except that the blasting process was done with fresh number 40-60 silicon carbide grit under 90 psi at a distance of 6 inches.

This surface (GB₂) was also prepared on all three alloys. The stainless steel was oxidized at 1,800° F for 20 minutes. The Inconel-X was oxidized at 2,000° F for 20 minutes, but the Inconel was oxidized at 2,000° F for periods of 20 minutes, 2 hours, 4 hours, and 8 hours.

Other Surfaces

Other oxidized surfaces were formed by combinations of the preoxidation treatments. For these surfaces the oxidation time for the Inconel and Inconel-X was 20 minutes at 2,000° F and for the 347 stainless steel was 20 minutes at 1,800° F. All combinations and the order of performing these treatments on the respective materials are listed in tables III, IV, and V.

RESULTS AND DISCUSSION

Inconel

The Inconel specimens tested in this study are listed in table III together with the preoxidation treatments, the oxidation times and temperatures, the total

normal emittances at 900°, 1,200°, 1,500°, and 1,800° F, and remarks on the physical conditions of the oxide surfaces. Six preoxidation treatments were used in investigating this alloy. These were, using symbols explained in table I, AR, E₁, GB₂, P, GB₂ followed by E₁, and GB₁ followed by E₁. The spectral normal emittance was measured for the AR, E₁, GB₂, and P specimens. The results for these four specimens are compared in figure 1 for 900° F and in figure 2 for 1,800° F.

Figures 1 and 2 show that the shapes of the spectral normal emittance curves for the E₁ and AR specimens are fairly similar. The curves for the GB₂ specimen were the highest and showed a progressive increase in emittance with increase in wavelength. The P specimen deviated from all others in this group by the fact that it tended to decrease in emittance with increasing wavelength and exhibited large maxima and minima. All specimens tended to increase in emittance, as shown from the total normal emittance values in table III, with increase in temperature.

Figures 3 and 4, for 900° and 1,800° F, respectively, are plots of spectral normal emittance for E₁, GB₂E₁, and GB₁E₁. They show the effects of prior grit blasting on the etched specimen and also compare the effects of the two grits used in the blasting process before etching.

The shapes of the spectral normal emittance curves for E₁ and GB₁E₁, figures 3 and 4, are almost identical, with the curve for the E₁ specimen being slightly higher. The curve for the GB₂E₁ specimen is the most irregular, with two strong maxima. A visual comparison of the surface roughnesses of the E₁ and GB₁E₁ specimens showed that the E₁ specimen is definitely rougher than the GB₁E₁ specimen. The etching tended to remove the peaks caused by the blasting process and to leave a very smooth surface. All specimens tended to increase in emittance, as shown in table III, with increase in temperature.

The standard oxidation time used in this study was 20 minutes. To investigate the change in the spectral normal emittance with change in oxidation time at the same temperature, 2,000° F, three GB₂ specimens were oxidized for longer periods of time, namely 2, 4, and 8 hours. These are designated as GB₂O₃, GB₂O₄, and GB₂O₅, respectively. The curves of spectral normal emittance are shown for these specimens and for the GB₂O₁ specimen in figures 5 and 6 for 900° and 1,800° F, respectively.

The curves for the GB₂O₁ specimen lie appreciably below the curves for the specimens with longer oxidation times, and it also has a different shape. The curves for the GB₂O₃, GB₂O₄, and GB₂O₅ specimens were almost identical. These specimens increased in emittance with increases in oxidation time, as shown in table II; however, there was no appreciable increase in emittance after 2 hours. The GB₂O₃, GB₂O₄, and GB₂O₅ specimens increase in emittance to 3 microns and then are almost constant, except for the minima at 6 microns, out to 14 microns

for the 900° F tests. The 1,800° F tests on these three specimens show the emittance to be extremely high from 1 micron to about 6 microns, beyond which they decrease and remain nearly constant at about 0.95 through 15 microns.

Inconel-X

The Inconel-X specimens tested in this study are listed in table IV, together with the preoxidation treatments, the oxidation times and temperatures, the total normal emittances for 900°, 1,200°, 1,500°, and 1,800° F, and remarks on the physical conditions of the oxide surfaces. Six preoxidation treatments were used in the studies of this alloy. These were, using symbols explained in table I, AR, E₁, GB₂, P, GB₁ followed by E₁, and GB₂ followed by E₁.

Spectral normal emittance curves for the first four of these oxide surfaces are compared in figures 7 and 8 for 900° and 1,800° F, respectively. The curves are very similar, except that the curves for the polished (P) specimen develop a series of pronounced maxima and minima at wavelengths greater than 6 microns. The emittance of all of these specimens increases with temperature, as shown in the total normal emittance values in table IV.

Figures 9 and 10, for 900° and 1,800° F, respectively, compare the spectral normal emittance curves for the E₁, GB₂, GB₂E₁, and GB₁E₁ specimens in order to show the effects of etching on the grit-blasted specimens.

The shapes of the spectral normal emittance curves of the GB₂, GB₂E₁, and GB₁E₁ specimens are very similar, but the curve for the etched specimen lies generally below the others and has pronounced irregularities beyond about 7 microns. The emittances of these specimens increase with temperature, and the curves become less irregular in shape at the higher temperature.

Stainless-Steel Type 347

The type 347 stainless-steel specimens tested in this study are listed in table V together with the preoxidation treatments, the oxidation times and temperatures, the total normal emittances at 900°, 1,200°, 1,500°, and 1,800° F, and remarks on the physical conditions of the oxide surfaces. Six preoxidation treatments were used in the investigation of this alloy. These were, using symbols explained in table I, AR, E₂, GB₂, P, GB₂ followed by E₂, and GB₁ followed by E₂.

Figures 11 and 12, for 900° and 1,800° F, respectively, compare the spectral normal emittances of the AR, E₂, GB₂, and P specimens. The curves for the AR, E₂, and GB₂ specimens have similar shapes, while the curves for the polished specimen, (P), deviates greatly from these by decreasing in emittance with increase in wavelength. It should be pointed out that the grit-blasted stainless-steel (GB₂) oxidized surface was definitely not reproducible. The curves shown in

figures 11 and 12 for one of the higher emittance samples, but it is not considered typical or representative. These specimens increase in emittance with increase in temperature.

Curves for the E_2 , GB_2 , GB_2E_2 , and GB_1E_2 specimens are compared in figures 13 and 14 for 900° and 1,800° F, respectively. These curves follow the expected trend with GB_2 having the highest emittance, GB_2E_2 next, and GB_1E_1 having the lowest emittance of the grit-blasted specimens. These specimens also increase in emittance with increase in temperature.

Special Tests

Figures 15, 16, and 17 provide information on the reproducibility of the spectral normal curves for similarly prepared specimens. Figure 15, which compares two samples of grit-blasted Inconel, shows a nearly perfect reproducibility. Figure 16, which compares two samples of etched stainless steel, shows a less perfect agreement; however, the average difference is less than 2 percent.

Most of these specimens follow the same spectral normal emittance curve as their duplicate specimen. The as-received (AR) oxidized Inconel specimens, when examined visually, looked grey-green in color and were slightly blotched. Since the coloration was somewhat uneven, it was considered that the spectral normal emittance curves would be most likely to show large variations from one sample to another. However, comparison of two specimens showed an average difference of only about 1 percent. This result led to tests of two additional samples prepared in the same manner. The extremes of these tests are shown in figure 17. The excellent agreement leads to the conclusion that an investigator cannot judge the emittance of the reproducibility of the infrared emittance spectrum by examining the specimen visually.

After the emittances were determined, adherence tests were performed by use of the Scotch tape test. This is a destructive test so one of the sample surfaces not chosen for testing was also used. The results of these tests are given in tables III, IV, and V. An oxide surface was considered to be adherent if, upon performance of the Scotch tape test, only a few small particles could be found on the tape and no noticeable damage was done to the specimen. If pieces of the oxide surface were pulled off by the tape and the portion of the surface that had been covered by the tape did not resemble the remainder of the specimen, then it was listed as a nonadherent oxide.

CONCLUDING REMARKS

This investigation has shown significant effects of preoxidation treatments and oxidation times on the spectral normal emittances of oxidized Inconel, Inconel-X, and type 347 stainless steel. In general, if a grit-blasted surface is etched before being oxidized, the final oxidized surface will have a lower emittance but will be more adherent and uniform. Of the two types of grit used in this study, the coarser grit provided the higher emittance. Polishing, even

to the degree used in this investigation, provided the lowest emittance of all specimens tested; possibly a high degree of polish, such as an electro-polished surface, would result in even lower emittance. In the one set of tests in which oxidation time was varied (Inconel), increasing oxidation time increased the emittance; increasing the time beyond 2 hours, however, produced no further effect.

For Inconel, the different preoxidation treatments and oxidation times resulted in total normal emittances ranging from 0.79 (polished) to 0.97 (grit-blasted, extended oxidation time) at 1,800° F. At 900° F, the values ranged from 0.64 to 0.88; and the spectral emittance curves indicate that at lower temperatures the variation would be even greater. It is concluded that, for accurate thermal-radiation calculations, it is not permissible to use emittance values obtained from sources that do not specify the exact surface preparation procedures as well as the test methods.

REFERENCES

1. Slomp, Wayne S., and Wade, William R.: A Method for Measuring the Spectral Normal Emittance in Air of a Variety of Materials Having Stable Emittance Characteristics. NASA, Presented at the Symposium on Measurement of Thermal Radiation Properties of Solids; Sept. 1962.
2. McMahon, H. O.: Thermal Radiation Characteristics of Some Glasses. Journal of American Ceramic Society, vol. 34, no. 3 (1951).

TABLE I.- PREOXIDATION TREATMENTS

Surface designation	Surface preparation
AR	This surface is prepared on materials as received from the supplier. The only treatment is a thorough degreasing. The specimens are swabbed with methyl alcohol, then with deionized water, then with reagent grade acetone, and finally rinsed with deionized water. The specimens are air dried.
E1	Degreased as for the AR specimens and then etched. The etching solution used was composed of 32 parts reagent grade nitric acid, 70 percent HNO_3 , 8 parts reagent grade hydrofluoric acid, 48 percent HF, and 32 parts deionized water. The etching time was $1\frac{1}{2}$ hours. The specimens were rinsed with deionized water, swabbed with methyl alcohol using cotton swabs, rinsed with deionized water again, and air dried.
E2	Same as E1 except the etching solution was composed of 100 ml of reagent grade hydrochloric acid, 36.5 to 38.0 percent HCl, 25 grams of powdered reagent grade chromic acid, 100 percent CrO_3 , and 50 ml of distilled water. The etching time was 3 to 5 min. The specimens were swabbed with cotton swabs in hot deionized water, then swabbed with methyl alcohol, rinsed with deionized water, and air dried.
GB1	The as-received stock was degreased with trichloroethylene in a vapor degreaser and then blasted with number 100 silicon carbide grit under 90 psi at a distance of 6 inches. The specimens were then degreased by the use of the vapor degreaser with trichloroethylene, as was done before blasting, and air dried.
GB2	Prepared the same as GB1 except that the stock was blasted with number 40-60 silicon carbide grit under 90 psi at a distance of 6 inches.
P	The specimens were degreased as for the AR specimens, and hand polished through four papers, 240, 320, 400, and 600 grit. They were degreased again in the same manner as the AR specimens and air dried.

TABLE II.- OXIDATION

Designation	Oxidation procedure		Remarks
	Time, min	Temperature, °F	
O ₁	20	2,000	Used in preparing Inconel and Inconel-X specimens
O ₂	20	1,800	Used in preparing the type 347 stainless-steel specimens
O ₃	120	2,000	Used for Inconel only
O ₄	240	2,000	Used for Inconel only
O ₅	480	2,000	Used for Inconel only

TABLE III.- OXIDIZED INCONEL

Specimen (*)	Preoxidation treatment (*)	Total normal emittance at temperature, °F				Other surface properties
		900	1,200	1,500	1,800	
ARO ₁	AR	0.64	0.68	0.76	0.80	Fairly adherent, reproducible, grey with small green blotches
E ₁ O ₁	E ₁	0.69	0.71	0.77	0.81	Very adherent, highly reproducible, grey-black
PO ₁	P	0.64	0.70	0.76	0.79	Fairly adherent, not very reproducible, green-grey
GB ₂ O ₁	GB ₂	0.77	0.80	0.85	0.87	Not very adherent or reproducible, grey-green
GB ₂ E ₁ O ₁	GB ₂ followed by E ₁	0.72	0.74	0.79	0.81	Rough, very adherent, highly reproducible, grey-black
GB ₁ E ₁ O ₁	GB ₁ followed by E ₁	0.68	0.72	0.78	0.80	Smooth, very adherent, highly reproducible, grey-black
GB ₂ O ₃	GB ₂	0.86	0.94	0.96	0.97	Adherent, reproducible, more green than the GB ₂ O ₁
GB ₂ O ₄	GB ₂	0.88	0.94	0.96	0.97	Adherent, reproducible, stable. The green color has disappeared leaving a grey-black oxide
GB ₂ O ₅	GB ₂	0.87	0.94	0.96	0.97	Very similar to GB ₂ O ₄

* Symbols explained in table I and table II.

TABLE IV.- OXIDIZED INCONEL-X

Specimen (*)	Preoxidation treatment (*)	Total normal emittance at temperature, °F				Other surface properties
		900	1,200	1,500	1,800	
ARO ₁	AR	0.82	0.84	0.86	0.88	Adherent, uniform, reproducible, grey-black
E ₁ O ₁	E ₁	0.80	0.82	0.84	0.87	Very smooth, adherent, uniform, reproducible, grey-black
PO ₁	P	0.80	0.82	0.86	0.89	Not very reproducible, light grey blotches
GB ₂ O ₁	GB ₂	0.85	0.88	0.90	0.92	Rough, adherent, reproducible, slightly blotched grey-black
GB ₁ E ₁ O	GB ₁ followed by E ₁	0.85	0.88	0.89	0.91	Very uniform, adherent, reproducible, grey-black
GB ₂ E ₁ O ₁	GB ₂ followed by E ₁	0.86	0.88	0.90	0.92	Very uniform, adherent, reproducible, grey-black

*Symbols explained in table I. and table II.

TABLE V.- OXIDIZED STAINLESS-STEEL TYPE 347

Specimen (*)	Preoxidation treatment (*)	Total normal emittance at temperature, °F				Other surface properties
		900	1,200	1,500	1,800	
ARO ₂	AR	0.82	0.86	0.89	0.91	Not very reproducible, nonuniform, blotched light grey
E ₂ O ₂	E ₂	0.84	0.87	0.90	0.91	Rough, uniform, adherent, reproducible, grey-black
PO ₂	P	0.74	0.77	0.81	0.83	Very smooth, uniform, adherent, reproducible, grey-black
GB ₂ O ₂	GB ₂	0.85	0.88	0.90	0.92	Not reproducible, nonuniform, blotched
GB ₁ E ₂ O ₂	GB ₁ followed by E ₂	0.81	0.83	0.86	0.88	Smooother than E ₂ O ₂ , uniform, adherent, reproducible, grey-black
GB ₂ E ₂ O ₂	GB ₂ followed by E ₂	0.84	0.86	0.88	0.90	Rougher than E ₂ O ₂ , uniform, reproducible, adherence not as good as GB ₁ E ₂ O ₂ , grey-black

* Symbols explained in table I and table II.

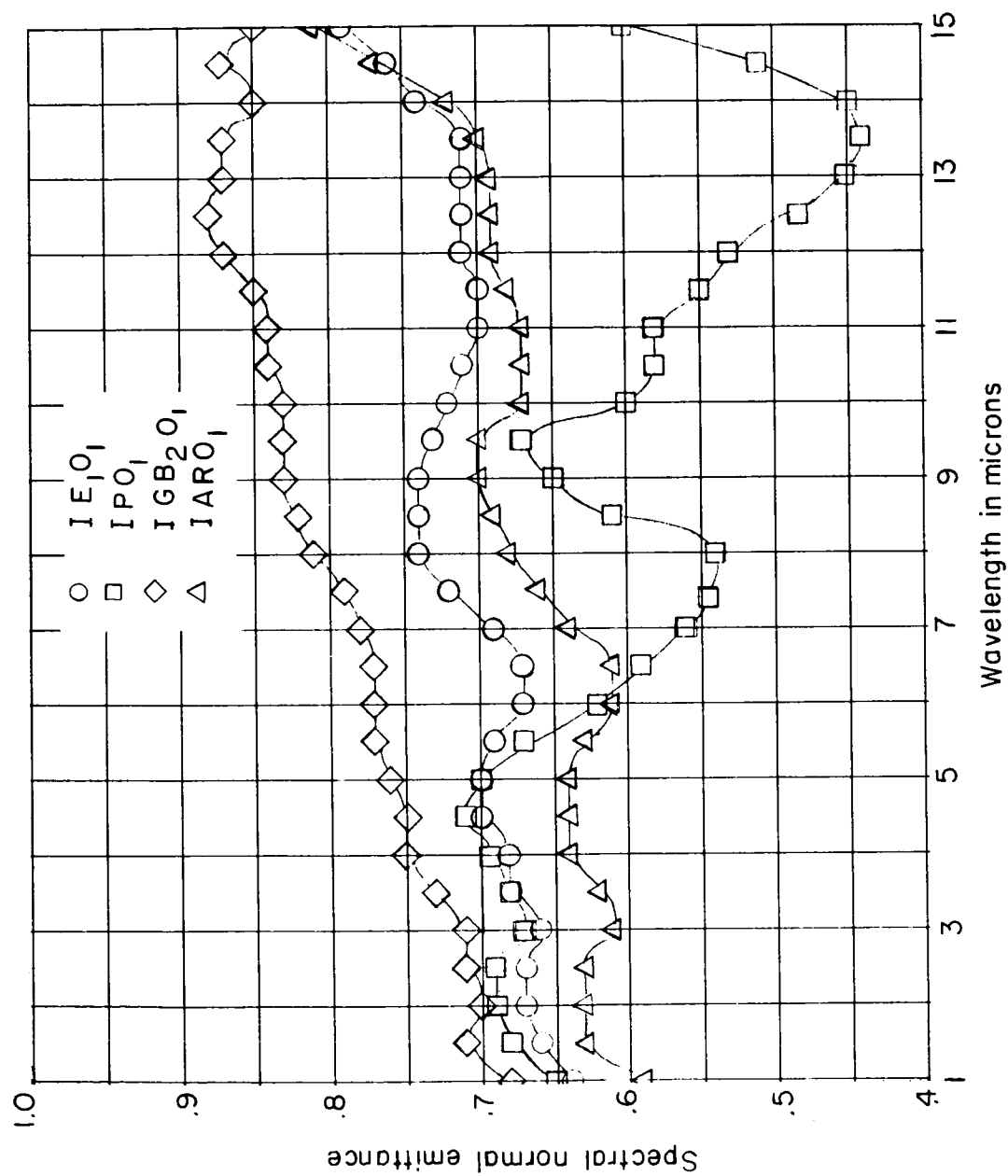


Figure 1.- Comparison of effects of preoxidation treatments on the spectral normal emittance of oxidized Inconel at 900° F.

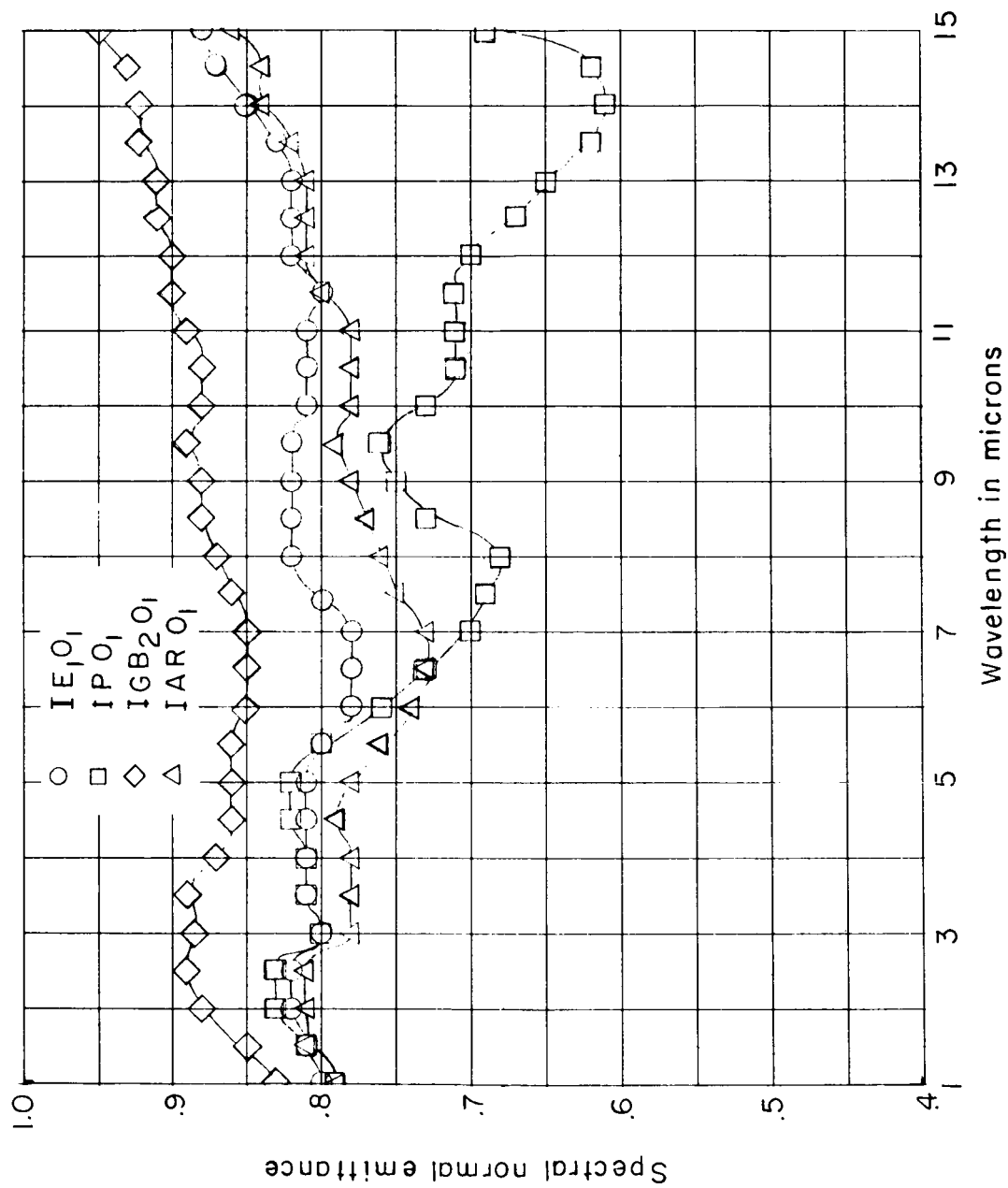


Figure 2.- Comparison of effects of preoxidation treatments on the spectral normal emittance of oxidized Inconel at 1,800° F.

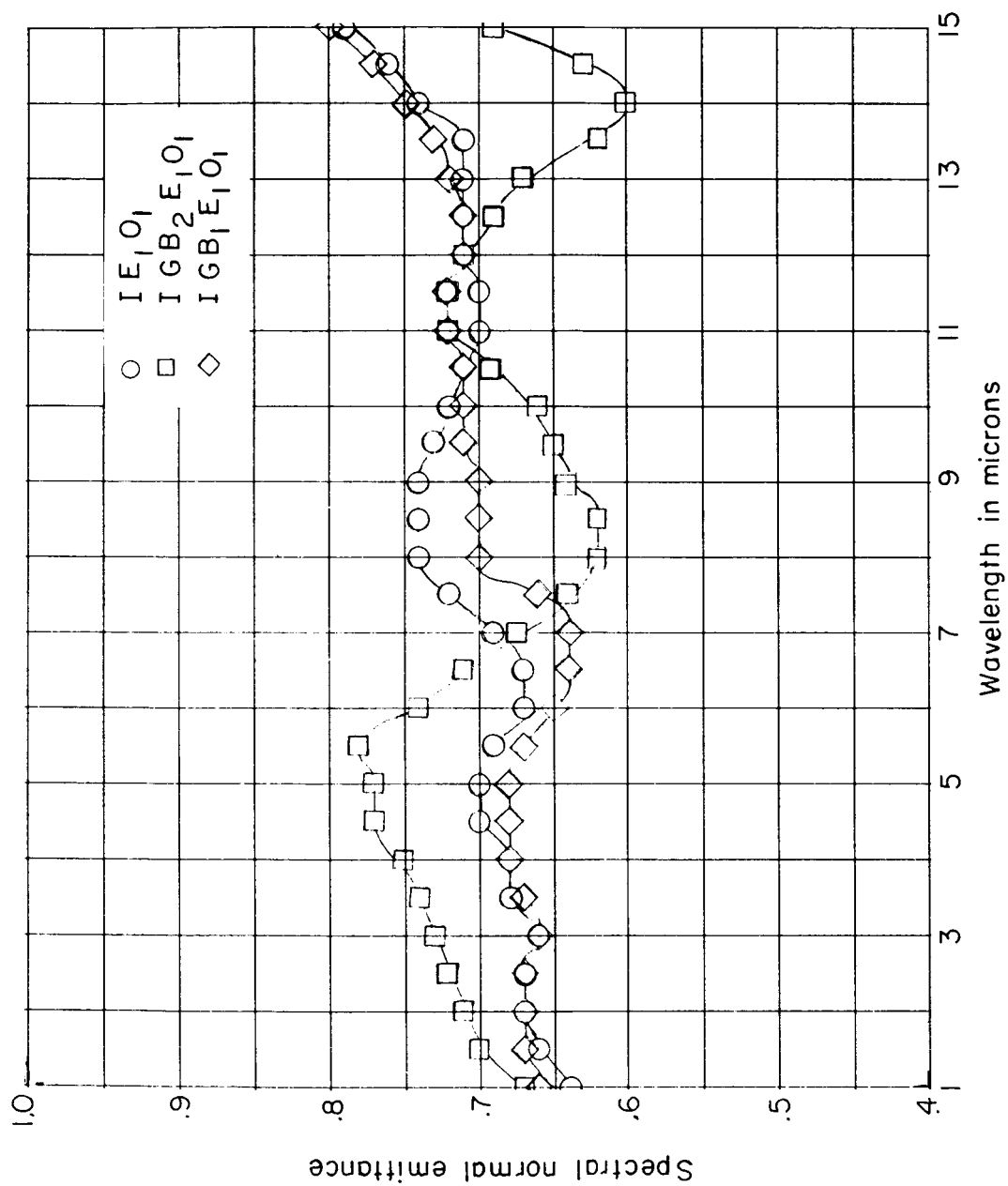


Figure 3.- Comparison of effects of combinations of preoxidation treatments on the spectral normal emittance of oxidized Inconel at 900° F.

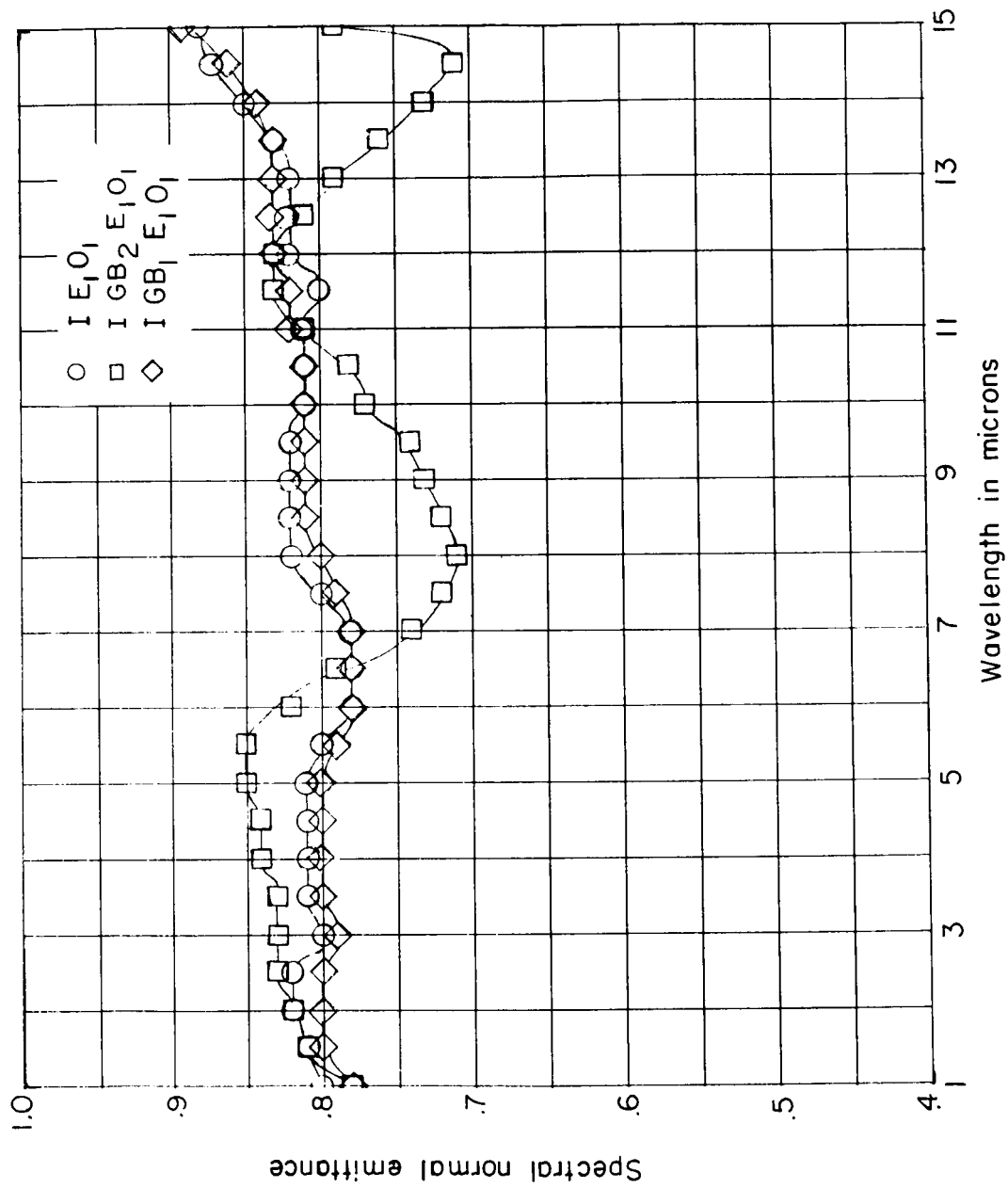


Figure 4.- Comparison of effects of combinations of preoxidation treatments on the spectral normal emittance of oxidized Inconel at 1,800° F.

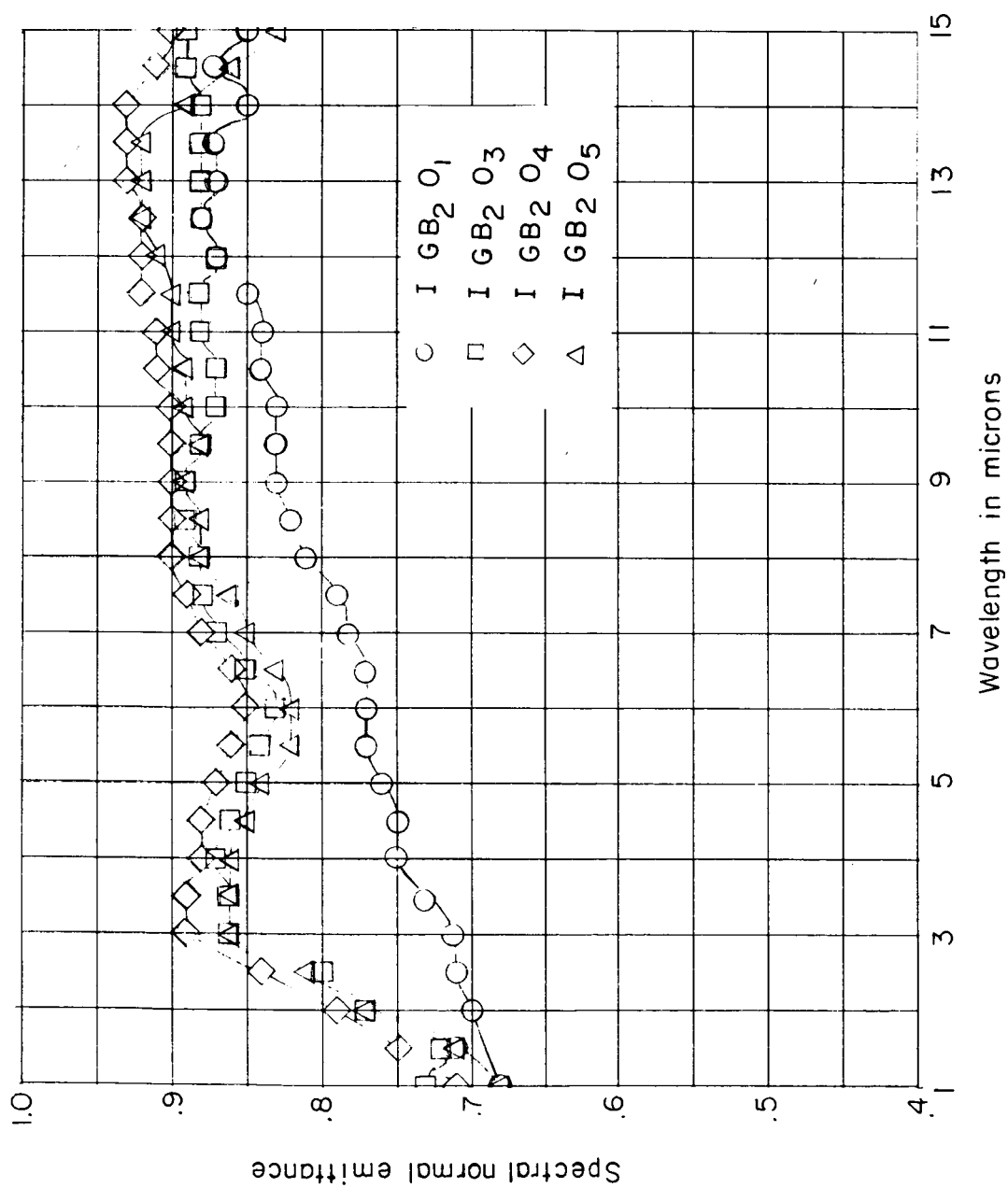


Figure 5.- Comparison of effects of oxidation time on the spectral normal emittance of oxidized Inconel at 900° F.

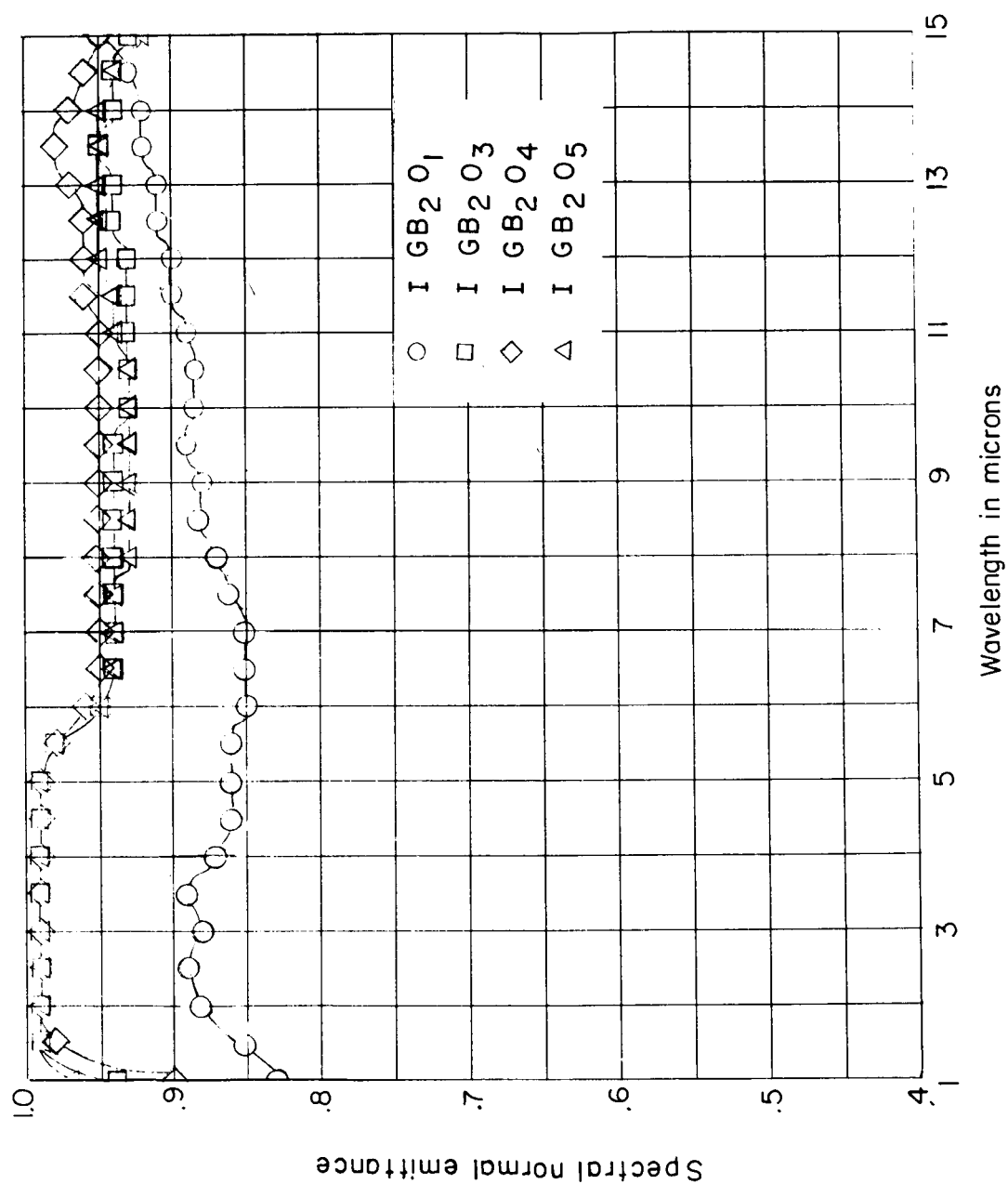


Figure 6.- Comparison of effects of oxidation time on the spectral normal emittance of oxidized Inconel at 1,800° F.

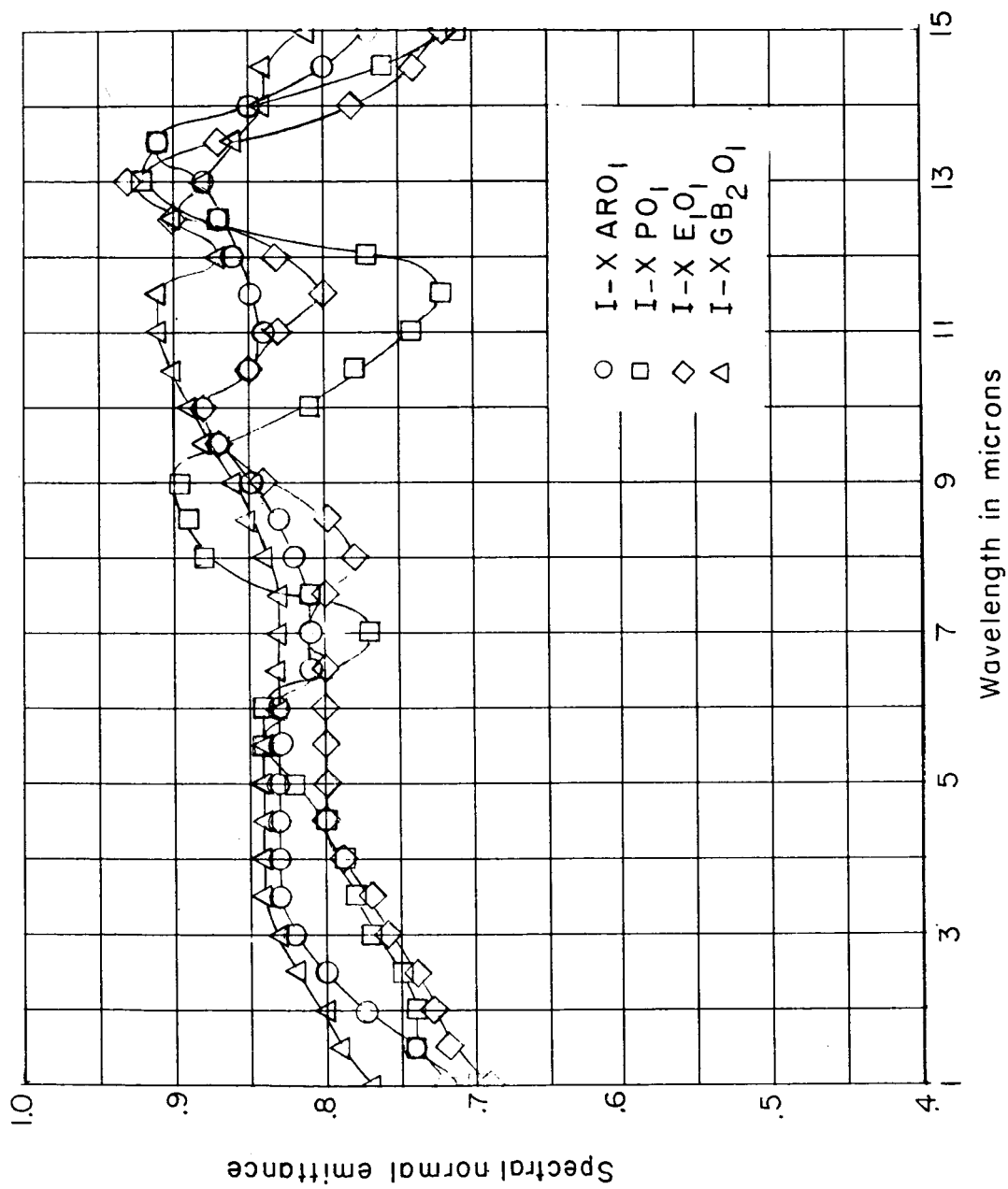


Figure 7.- Comparison of effects of preoxidation treatments on the spectral normal emittance of oxidized Inconel-X at 900° F.

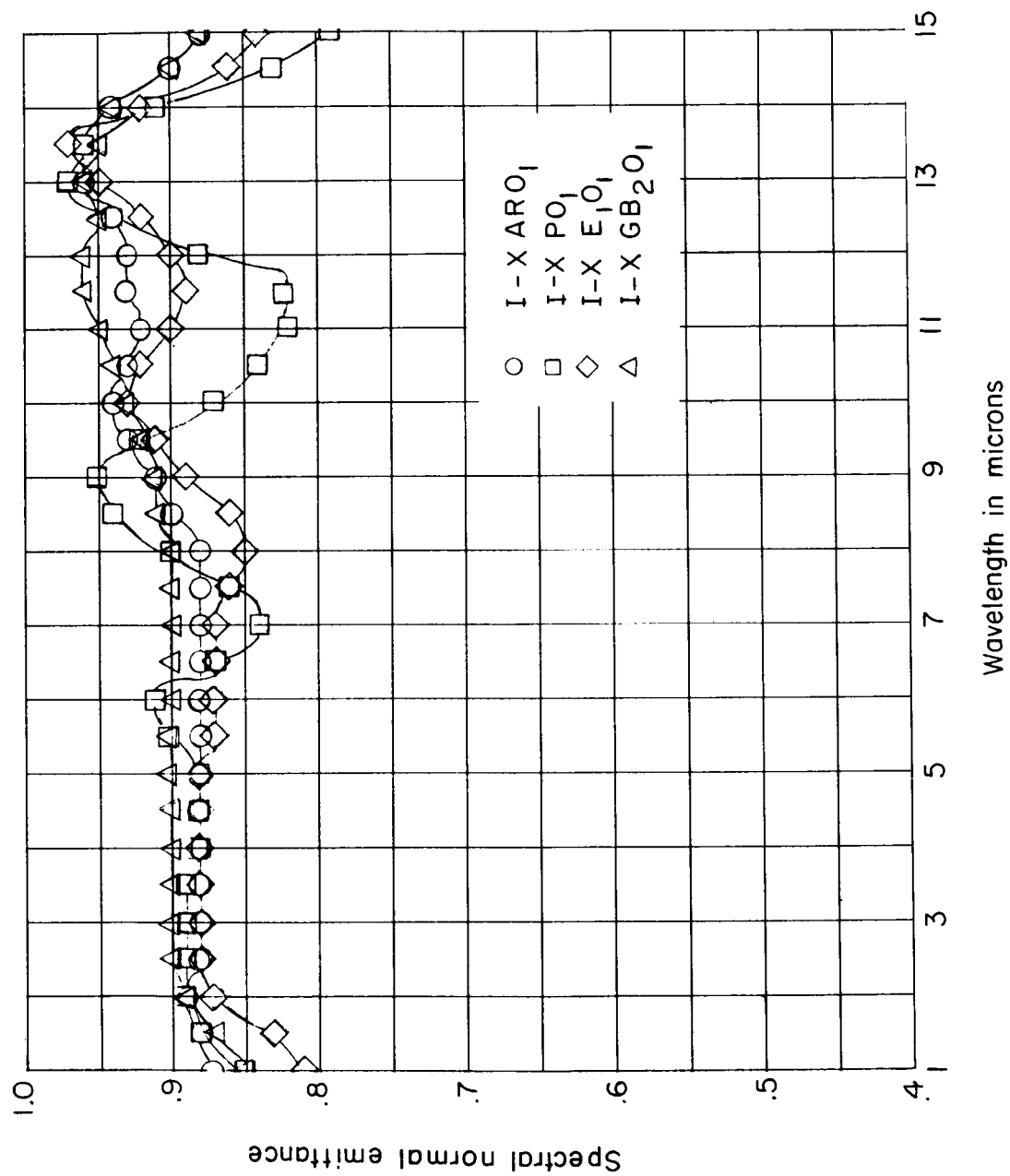


Figure 8.- Comparison of effects of preoxidation treatments on the spectral normal emittance of oxidized Inconel-X at 1,800° F.

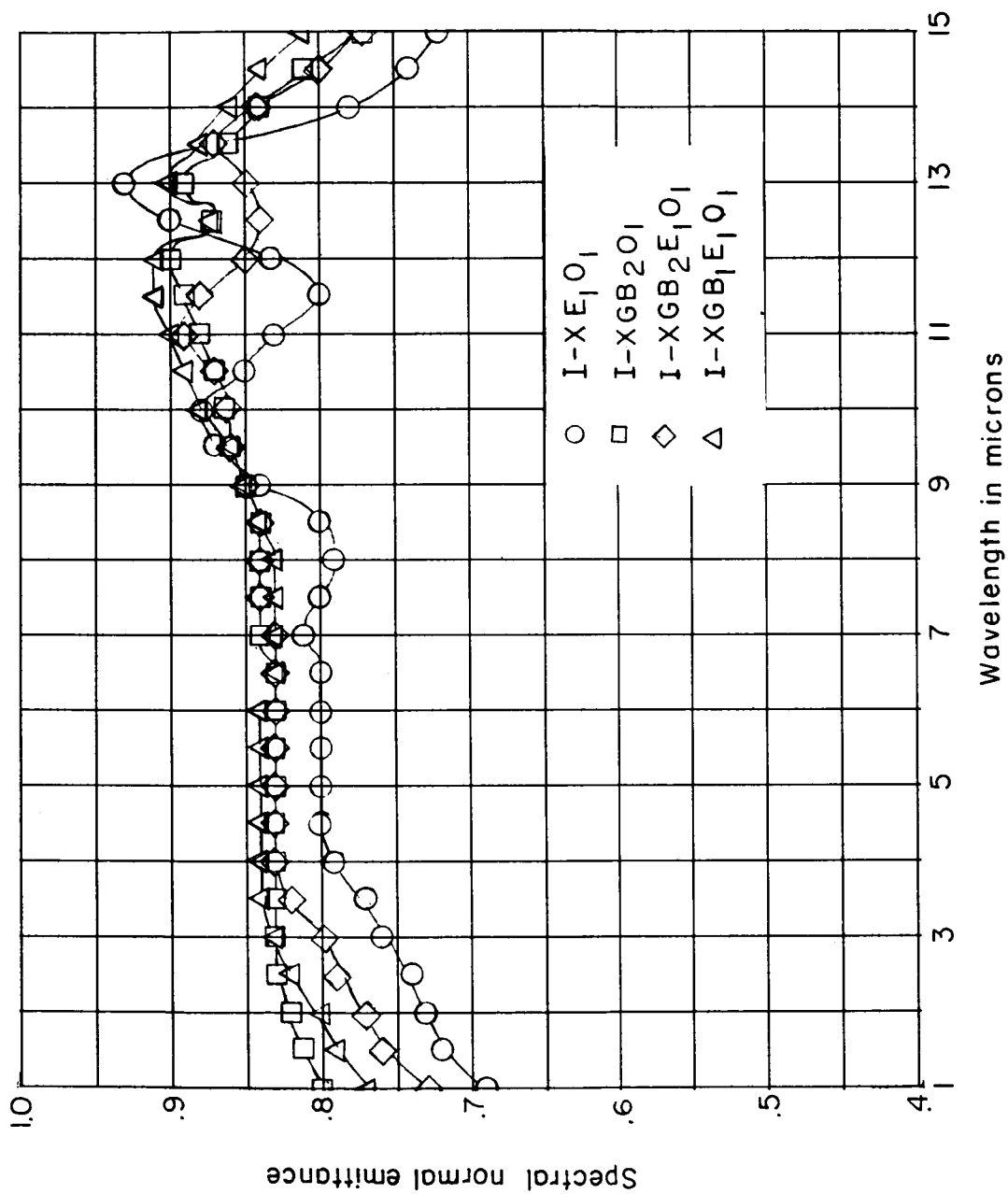


Figure 9.- Comparison of effects of combinations of preoxidation treatments on the spectral normal emittance of oxidized Inconel-X at 900° F.

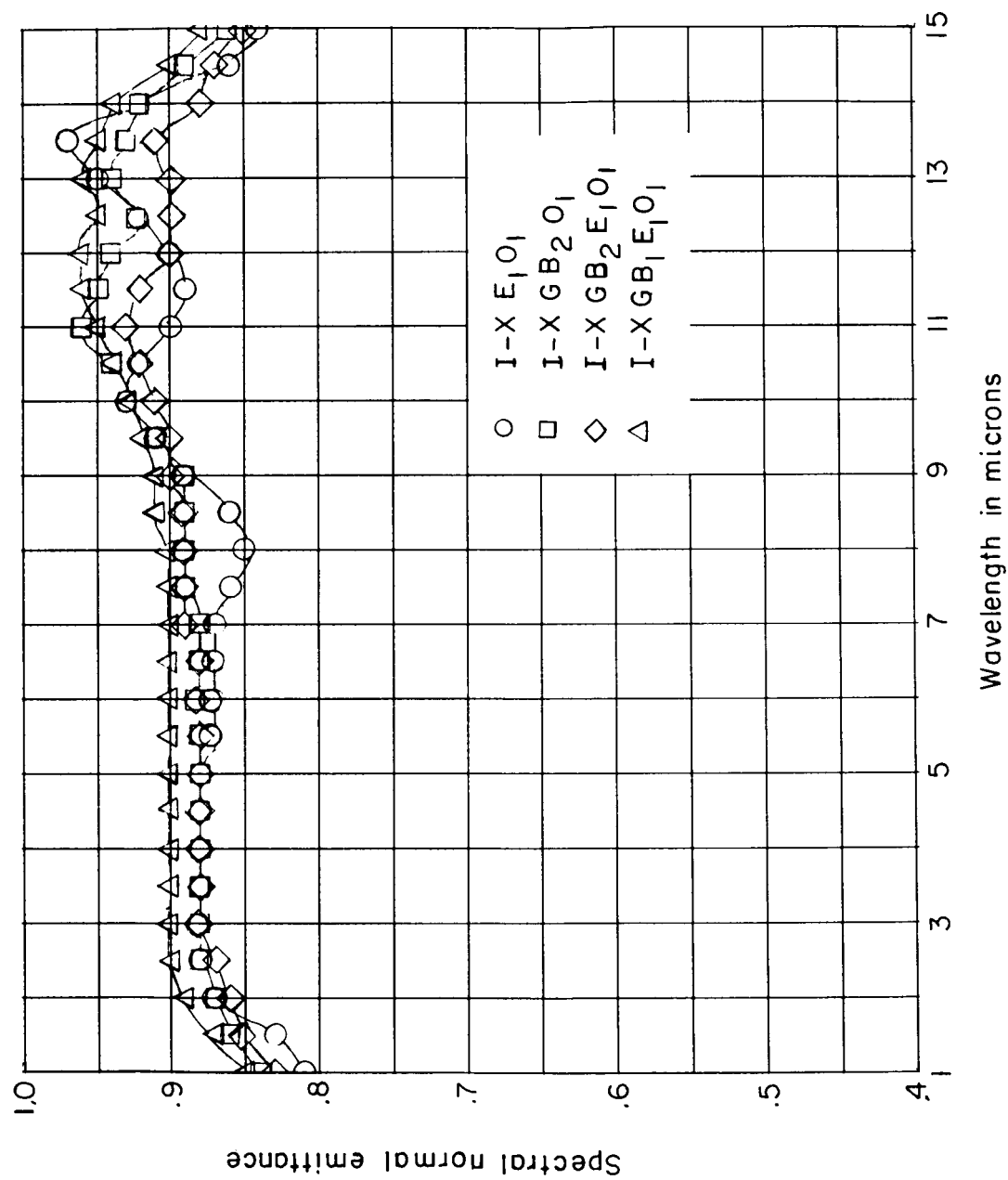


Figure 10.- Comparison of effects of combinations of preoxidation treatments on the spectral normal emittance of oxidized Inconel-X at 1,800° F.

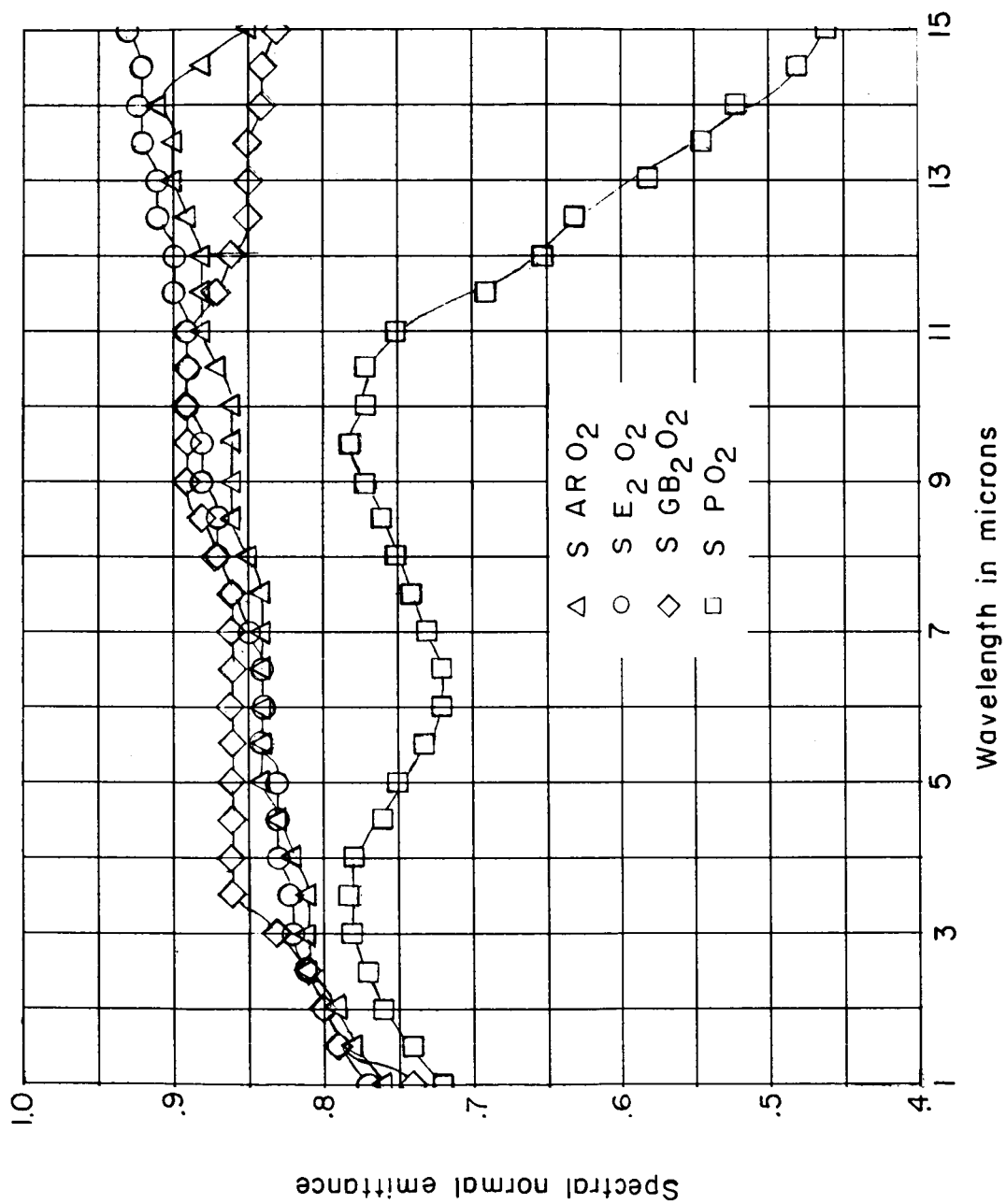


Figure 11.- Comparison of effects of preoxidation treatments on the spectral normal emittance of oxidized type 347 stainless steel at 900° F.

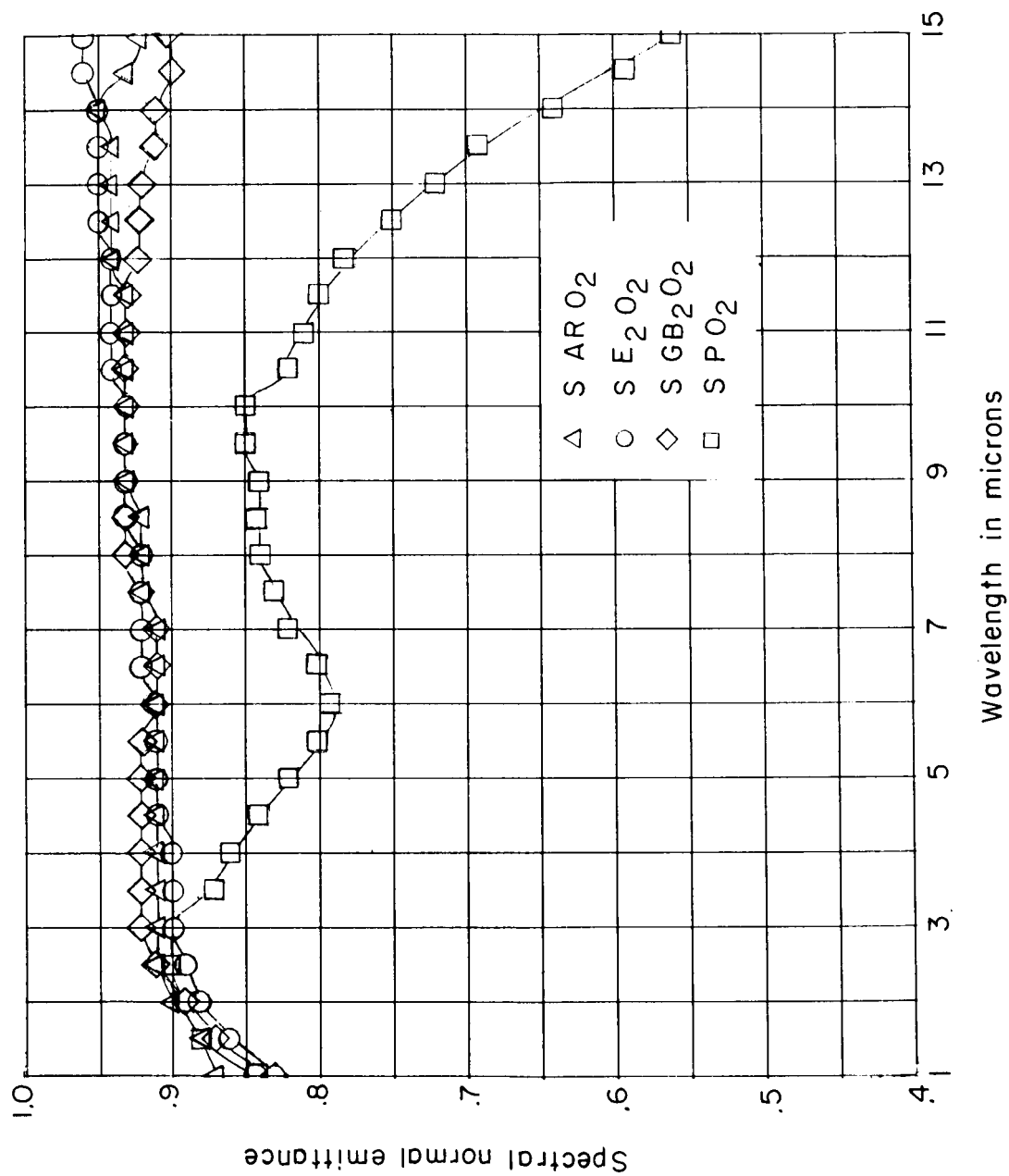


Figure 12.- Comparison of effects of preoxidation treatments on the spectral normal emittance of oxidized type 347 stainless steel at 1,800° F.

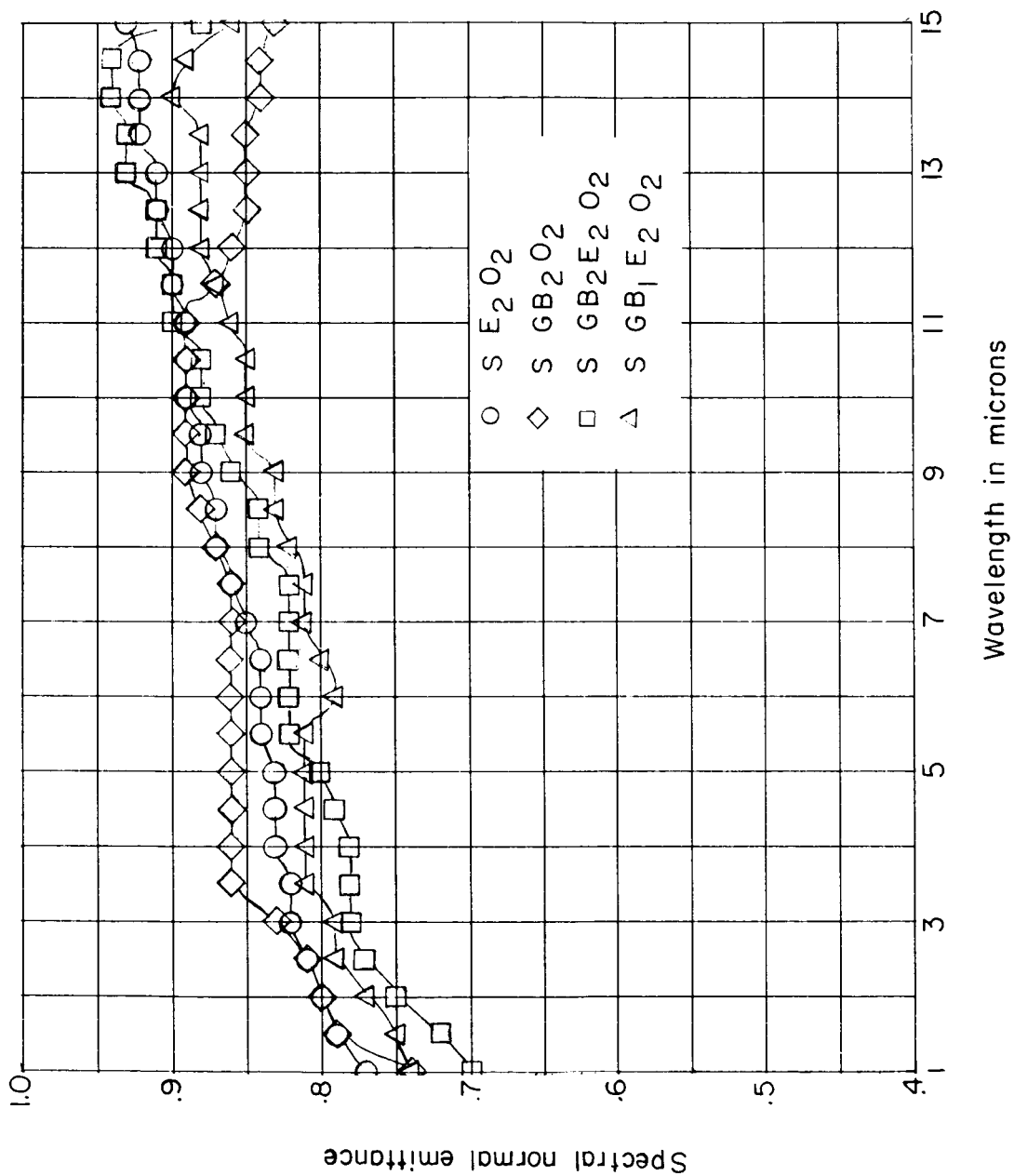


Figure 13.- Comparison of effects of combinations of preoxidation treatments on the spectral normal emittance of oxidized type 347 stainless steel at 900° F.

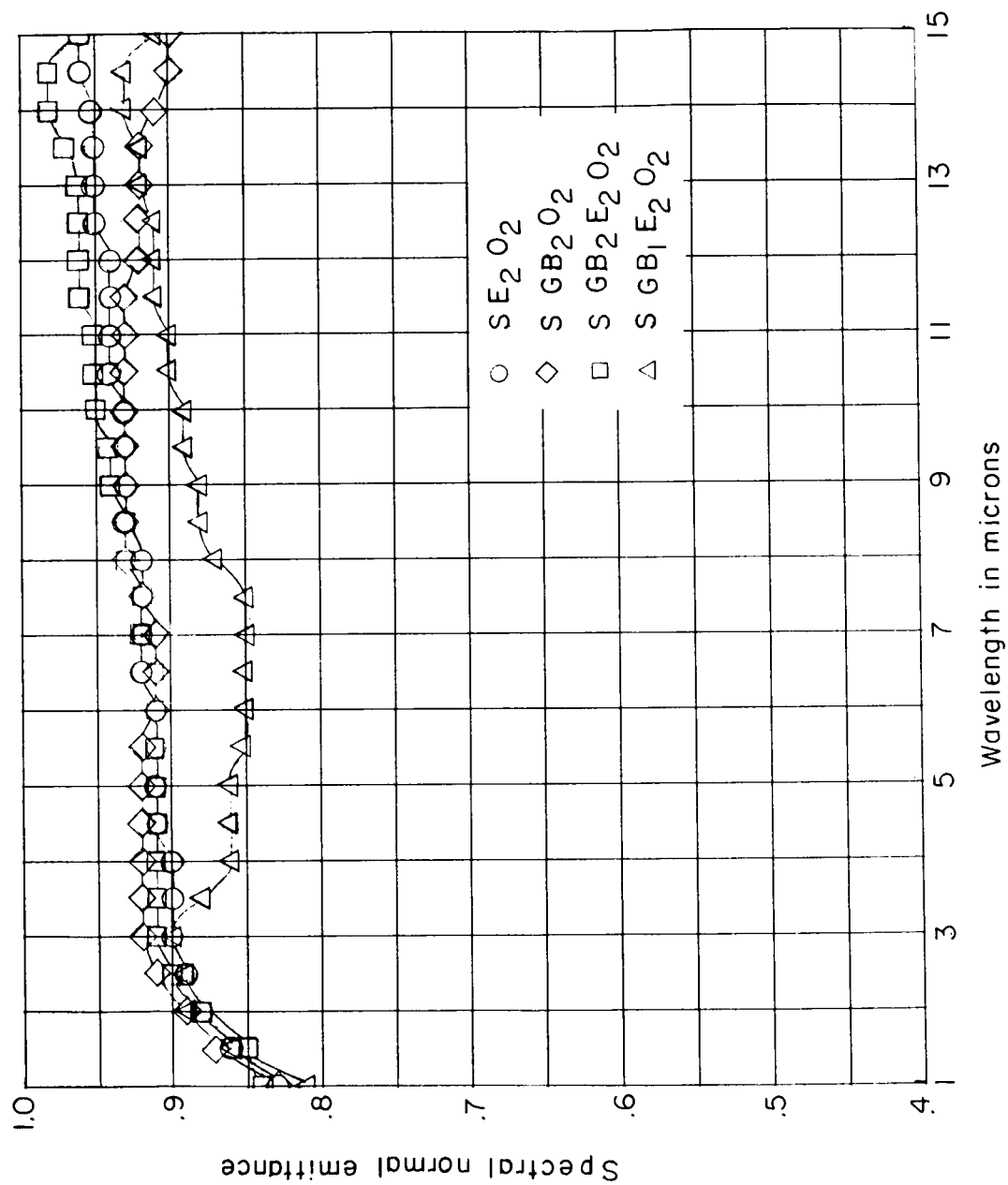


Figure 14.- Comparison of effects of combinations of preoxidation treatments on the spectral normal emittance of oxidized type 304 stainless steel at 1,800° F.

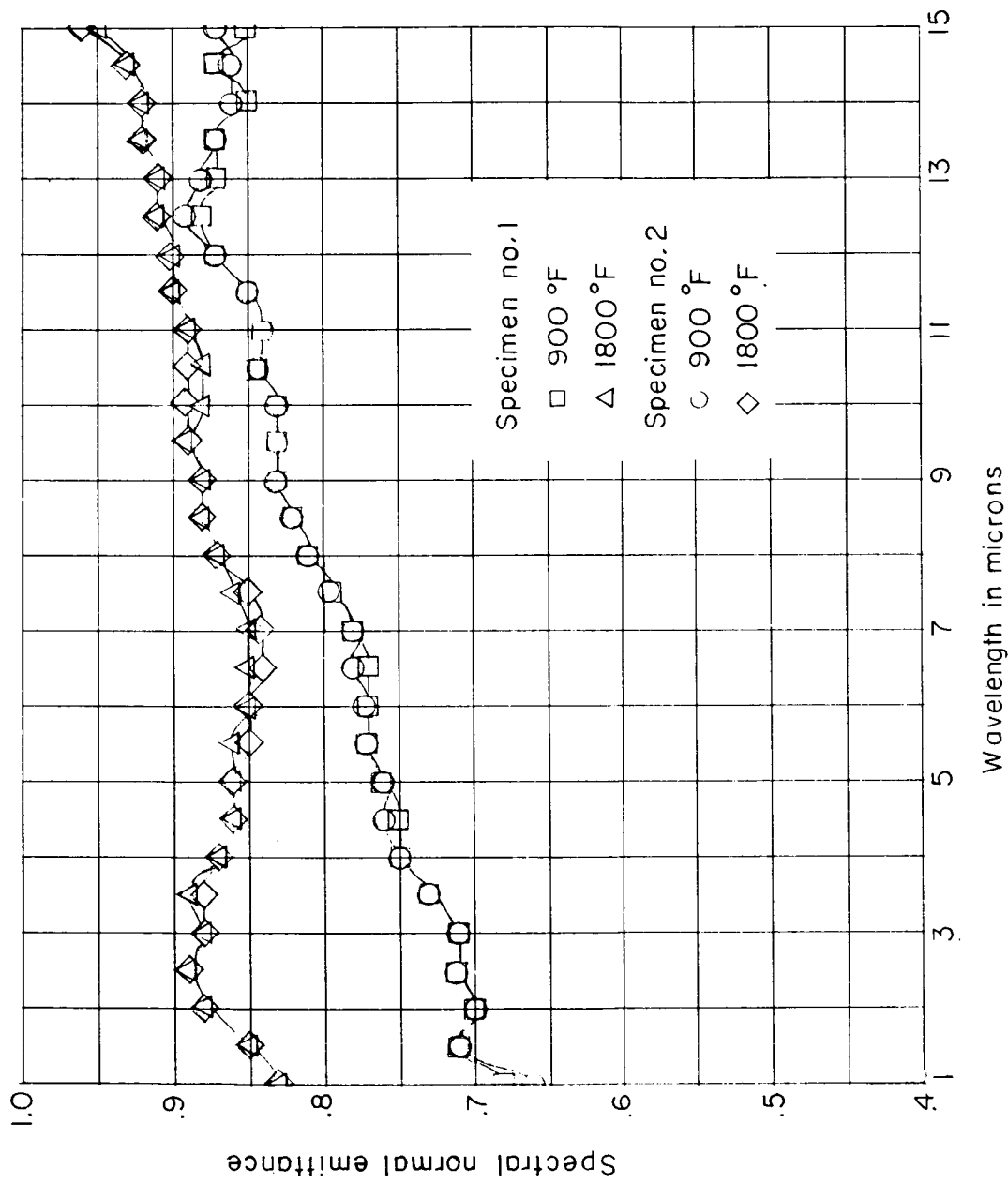


Figure 15.- Reproducibility of spectral normal emittance curves. Curves for two oxidized grit-blasted Inconel specimens (I GB20₁), prepared and tested by the same procedure.

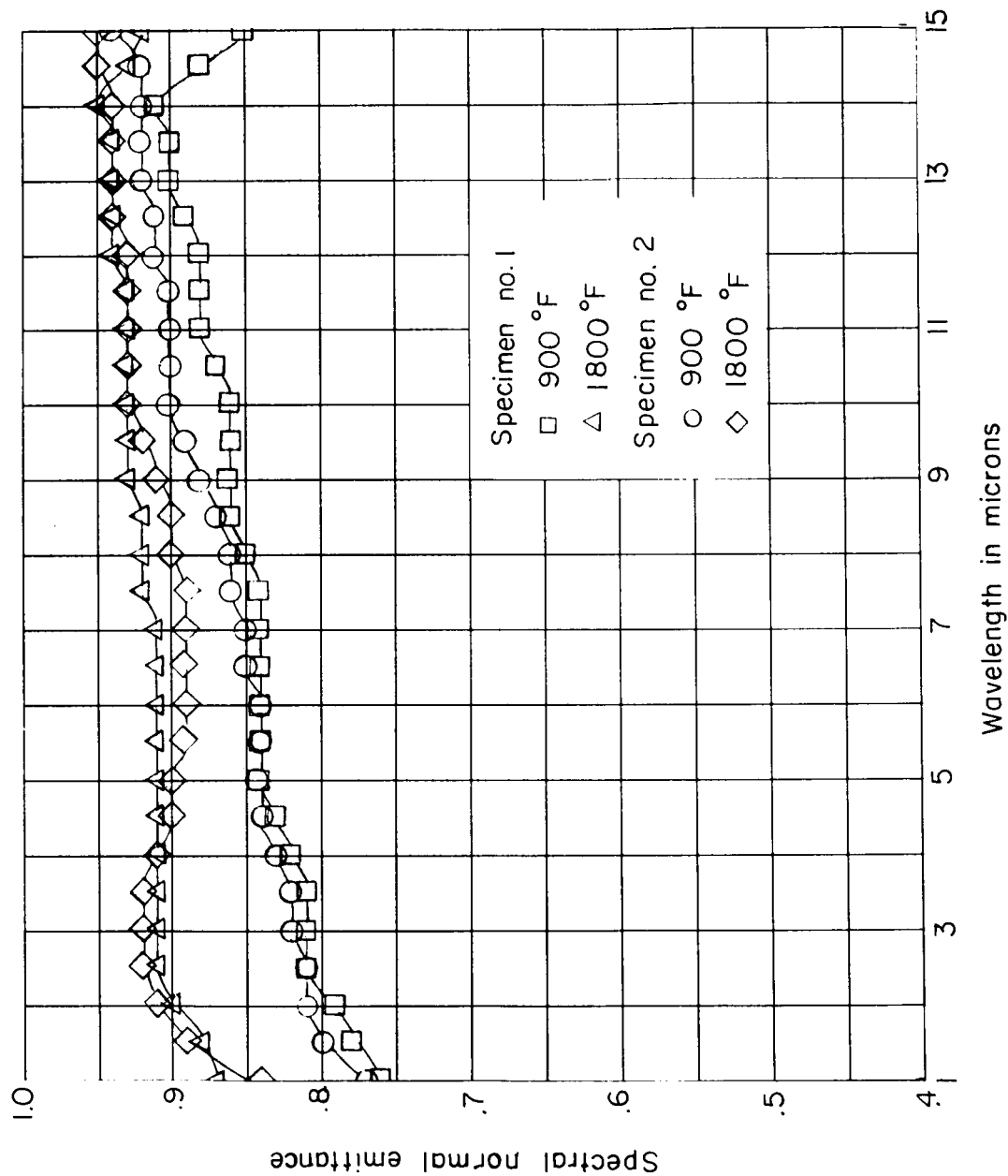


Figure 16.- Reproducibility of spectral normal emittance curves. Curves for two etched oxidized type 347 stainless-steel specimens ($S E_2O_2$), prepared and tested by the same procedure.

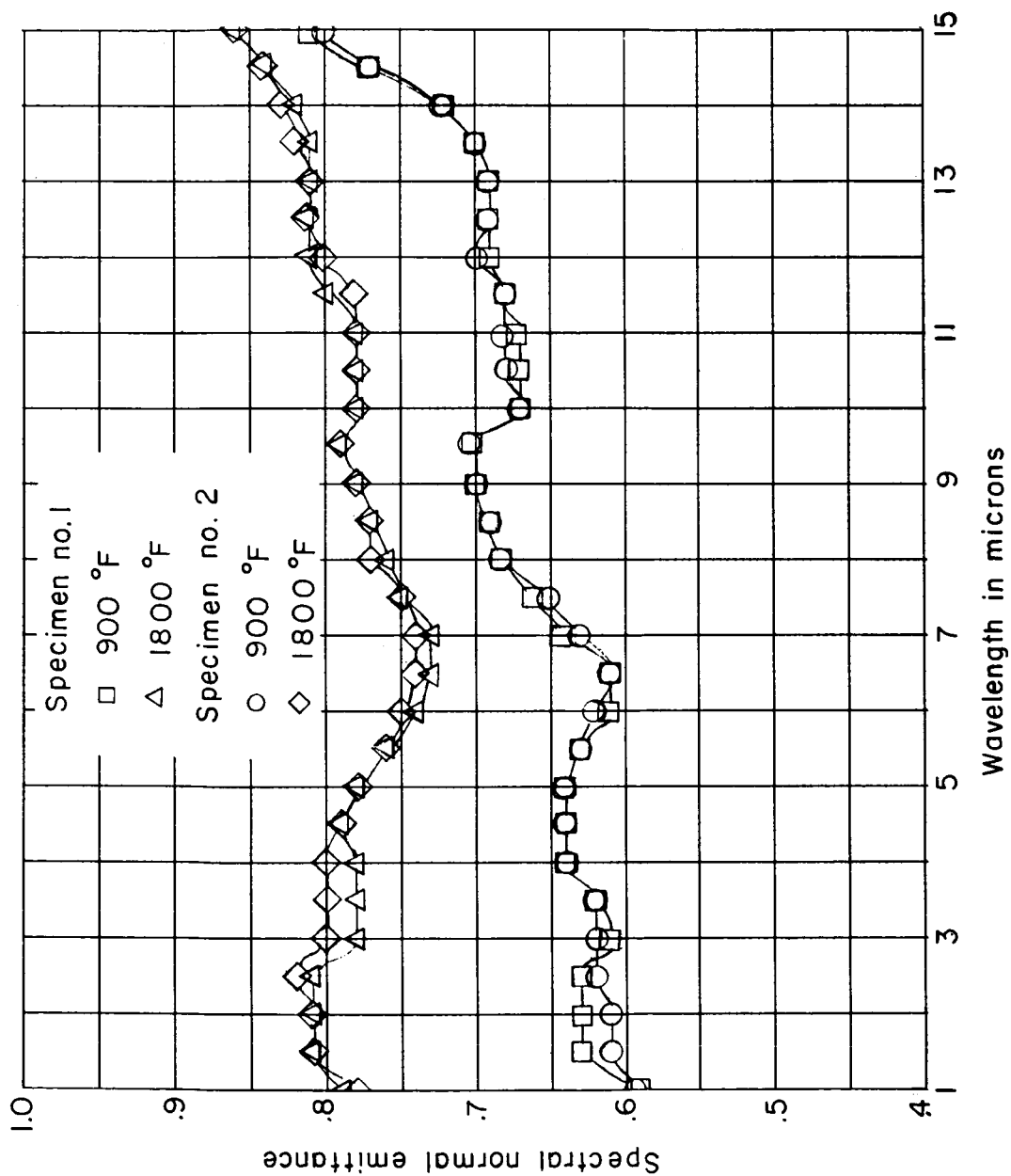


Figure 17.- Reproducibility of spectral normal emittance curves. Curves for two specimens representing the maximum deviation of four as-received oxidized Inconel specimens (I AR 0₁), prepared and tested by the same procedure.